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Off-the-Shelf Antifreeze Admixtures

Charles J. Korhonen

April 2002

Abstract: Cold weather places serious constraints on today's concreting operations. As temperatures drop, concrete sets more slowly, takes longer to finish, and gains strength less rapidly. If temperatures dip too low, the risk is that the mixing water will freeze, leading to irreparable damage. Current guidance limits cold-weather protection of fresh concrete to insulation, supplemental heating, and temporary shelters to keep the concrete at or above 5°C throughout the curing process. This paper studies the use of commercial admixtures in combination with one another to depress the freezing point of the mixing water and to allow the concrete to gain strength at below-freezing temperatures without thermal protection.

Technical Report
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Prepared for
OFFICE OF THE CHIEF OF ENGINEERS

PREFACE

This report was prepared by Charles J. Korhonen, Research Civil Engineer, Civil Engineering Research Division, U.S. Army Engineer Research and Development Center (ERDC), Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire.

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Off-the-Shelf Antifreeze Admixtures

CHARLES J. KORHONEN

1 INTRODUCTION

Of the many admixtures that are available for use in concrete today, none are available that can protect fresh concrete from freezing. Admixtures are available that allow concrete to gain strength when the air temperature is below freezing, but even when used at their highest recommended doses, they provide minimal protection when the concrete temperature is below freezing. They protect concrete by accelerating cement hydration to increase the rate of internally generated heat so that the concrete might be kept warm long enough to gain needed strength before freezing occurs. The extent of this protection depends on how quickly heat is evolved within the concrete and how quickly it is lost to the outside environment. The problem is that individual admixtures are not dosed at high enough concentrations to depress the freezing point of fresh concrete by more than a degree or two. Consequently, freshly placed concrete has to be thermally protected until it has cured.

Because extra large doses of chemicals—the amounts needed to protect concrete at subfreezing temperatures—are believed to be harmful to concrete in some way (ACI 2000), admixture-laden concrete has not been promoted in the United States. However, studies done elsewhere in the 1950s show that large doses of chemicals can prevent fresh concrete from freezing without causing detrimental side effects to the final product (Korhonen 1990). More recently, data from laboratory testing done in the United States in the early 1990s show that chemicals, some not normally used in U.S. admixtures and at doses two to three times higher than those used by other chemicals in U.S. admixtures, allowed fresh concrete to gain strength when the temperature of the concrete was nearly −20°C (Korhonen and Cortez 1991; Korhonen et al. 1991; Korhonen et al. 1992a, b; Korhonen et al. 1994). These findings eventually led to the development in 1996 of this country's first prototype commercial admixture capable of protecting concrete down to –5°C (Korhonen and Brook 1996, Korhonen et al. 1997). However, as with other technological advances, the transition of these

findings into practice has been stymied by the lack of acceptance standards and by a wariness of many to be the “first” to try a new technology. Tort liability concerns discourage manufacturers from producing new materials that are not supported by industry standards, and others shy away simply because the industry has become adept at finding other work to do during the winter. Follow-on studies done by the U.S. Army Corps of Engineers for the Tennessee Valley Authority (TVA) (Korhonen et al. 1998) in 1997 showed that the lack of standards could be overcome by formulating an antifreeze admixture from individual admixtures already approved for everyday use in concrete and that, with a little additional work, a true antifreeze admixture might be possible using existing technology. The Corps showed that by using ordinary admixtures, TVA was able to place concrete at –8°C air temperatures without obtaining special approval for the admixtures. However, problems with maintaining a workable mix over extended times meant that further work was needed to render this approach practical for general construction. To support the development of a new generation of admixtures, the Civil Engineering Research Foundation spearheaded an effort in October 2000 to develop a draft of what is hoped may eventually serve as the basis of an industry standard for antifreeze admixtures (Korhonen and Ryan 2000).

Although standards are ultimately needed to open the door to widespread use of antifreeze admixtures, it could be a long wait as creating, approving, and accepting them can take years. Thus, rather than having to wait for new standards to be developed before this technology can be used, this paper reports on the continuation of the TVA effort to develop antifreeze admixtures from the many admixtures that are routinely used in concrete today.

2 CONVENTIONAL PRACTICE

Cold-weather concreting practice has remained unchanged since the 1930s. The concrete ingredients must be heated, the substrate must be thawed, and the concrete itself must be kept warm while it cures. This section describes how fresh concrete behaves in the cold and how current construction practice accommodates this behavior. It also provides a useful contrast to the method of using antifreeze admixtures presented later in this paper.

Effect of temperature on time of setting

The rate at which concrete stiffens, or sets, is important to construction because stiffness determines the workability of the concrete, that is, whether it can be properly placed and consolidated. Concrete is considered to be workable as long as it can be made mobile by vibration. As soon as concrete no longer responds to a vibrator, it has reached initial set. By this time workers should have completed floating the concrete and begun to trowel it. As stiffening continues, the concrete reaches final set, where troweling becomes very difficult, if not impossible, and measurable strength gain begins. ASTM C 403 (ASTM 1989a) defines a method used by laboratories to measure the elapsed time, after water is first added to the cement, for the mortar fraction of concrete to reach initial and final set.

Setting is caused by a chemical reaction between cement and water that, when sufficiently completed, binds the aggregate together, giving concrete its strength. Neville (1988) reports that both initial and final setting times increase as temperatures decrease (Fig. 1). Though an industry rule-of-thumb suggests that initial setting times double for each 10°C drop in temperature, Neville's data show this factor to be closer to 1.5 times throughout a significant range of temperatures. For example, the 4.25-hour initial setting time at 20°C increased 1.5 times to 6.4 hours when the mortar was cured at 10°C. In the case of final setting, mortar behaves similarly until the temperature dips below 10°C. At about that point the multiplication factor increases to roughly 2.5. Although final setting is the one most affected by low temperatures, initial setting is more important when finishing is the concern. That is because a mix that sets too slowly causes the finishing crew to wait longer, delaying progress and increasing the cost of construction. Slow setting is also a concern when placing concrete on a sloped surface. Conversely, concrete that sets too rapidly can present a problem when travel times to the job site are great.

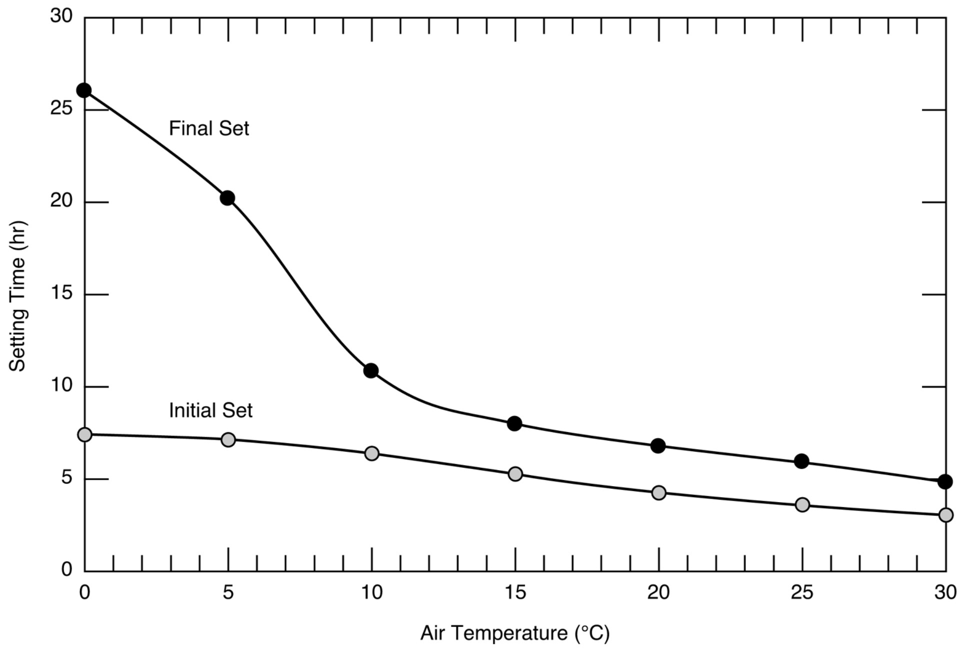

Figure 1. Relationship between setting time and temperature.

To alleviate the slowness of setting caused by low temperatures, the mix design of concrete can be altered in several ways: changing the type of cement, using a finer cement, using more cement, or decreasing the water–cement ratio. Appropriately altering one or more of these factors can increase both the rate and the amount of heat developed at early age to speed up the setting process. Table 1 provides an example of how changing these factors can affect the initial setting time of concrete.

Table 1. Initial setting times (hr) of concrete containing 307 kg/m^3 of portland cement (Dodson 1994). Unless noted, all data are derived using Type I cement with samples cured at room temperature.

Cement		Surface Area		Amount		Water/Cement	
Type	Hr	m^2/kg	Hr	Sacks	Hr	Ratio	Hr
I	4.0	435	3.6 hr	3.75	4.8 hr	0.58	5.2 hr
II	4.8	322	4.0	5.9	3.9	0.54	4.7
III	3.3	556	2.8	—	—	—	—

For example, of the three cement types presented in Table 1, changing from Type I to Type III cement causes the hydration rate to increase and, in turn, the

setting time to decrease by about 1.2 times (i.e., from 4.0 hours to 3.3 hours), which nearly compensates for the delay caused by the 10°C drop in temperature mentioned previously. Changing from Type I to Type II cement, on the other hand, has the opposite effect. It increases the setting time by 1.2 times, which, in effect, causes the concrete to behave as if it were nearly 10°C cooler than it actually is. For this reason, Type II cement is not usually recommended for normal winter concreting where the combined effect of a slow-setting cement and low temperatures could seriously delay construction schedules.

In the case of cement fineness, changing the surface area of cement, according to Table 1, can be even more effective than using Type III cement. Specifically, the finest cement (556 m^2/kg) decreased the initial setting time by 1.3 times compared to the coarser ground cement (435 m^2/kg).

The final two factors that can be altered—increasing the amount of cement and decreasing the water–cement ratio—also speeds up setting times, though not as dramatically as did changing cements or using a more finely ground cement. For example, Table 1 suggests that an extra sack of cement decreases initial setting by 1.1 times while it takes a 0.04 drop in w/c ratio to effect the same change. Though the effect of altering the mix design in more than one way simultaneously is not illustrated in Table 1, it can be imagined that using extra-fine Type III cement along with a low w/c ratio could be enough to compensate for more than a 10°C temperature drop. These changes are enough to speed up construction during cool weather, but they are not enough to preclude the need for thermal protection during subfreezing weather.

Another factor that is often used to speed up the hydration rate of cement, not shown in Table 1, is to use chemical additives called accelerators. These chemicals, according to ASTM C 494 (ASTM 1997a), decrease the initial setting time of concrete by at least one hour but by no more than 3.5 hours with respect to reference concrete cured at 23°C. With respect to the mortar made with the Type I cement in Table 1, a one-hour reduction in the time of setting means that the accelerator would cause the mortar to behave approximately as if it was made with Type III cement. The 3.5-hour limit set by ASTM is more of practical significance than it is a technical limitation because concrete that sets faster than this limit is probably unusable to everyday construction operations. There just would not be enough time to discharge the concrete from the truck, let alone finish it properly, before it stiffened. Although accelerating admixtures, when used within recommended doses, shorten the duration of cold-weather protection, the freezing point of the concrete is not much lower than that of admixture-free concrete, so thermal protection is still needed during curing.

Effect of temperature on strength gain

The rate at which concrete gains strength also is important because it dictates how quickly forms can be stripped and reused, which, like setting, can control the pace of construction. Strength gain is a function of the degree to which the cement has hydrated, and the rate at which concrete gains strength is temperature-dependent. Within the range of temperatures usual to winter and summer concreting situations, it is generally observed that the higher the temperature is, the more rapid is the strength gain. This relationship invariably leads to the conclusion that hot weather is the best time to cast concrete because concrete sets up and gains strength faster when it is hot than when it is cold. Figure 2 supports this contention by showing that concrete gains 25% more strength in three days when held at 40°C than it does when held at 20°C and more than doubles the amount of strength gained at 5°C. Getting extra early-age strength can shave days off a project's schedule, especially when forms have to be reused multiple times. For a contractor, this is good news. However, the strength benefits are short-lived as, by day seven, the early age strength increase caused by the high temperature vanishes. For two identical concretes cured for seven days, the one cured at 40°C attained 76 percent of its potential strength while the other one cured at 20°C attained 80 percent. Moreover, the concrete, at 28 days, ends up 15% weaker when cured at 40°C than when cured at 20°C. To the owner of a structure built during hot weather, this is not good news. Thus, though hot weather might allow faster-paced construction, it might also result in weaker, less durable concrete.

Cold weather, on the other hand, can be beneficial when it comes to long-term strength gain. At early ages, however, cold weather slows down the pace of construction by causing strengths to develop slowly. To stay out of trouble, one must be aware of this delay and allow for longer curing times. For example, Figure 2 shows that concrete cured for three days at 5°C is 70 percent weaker than reference concrete cured the same time at 20°C, and it is even weaker when cured at 0°C. However, provided ice does not form inside the concrete, cold weather produces stronger concrete in the long run—up to 20% stronger in the case of concrete cured at 5°C for 56 days and then warmed back up to room temperature (Korhonen and Orchino 1999). Cold weather can be the best time to place concrete.

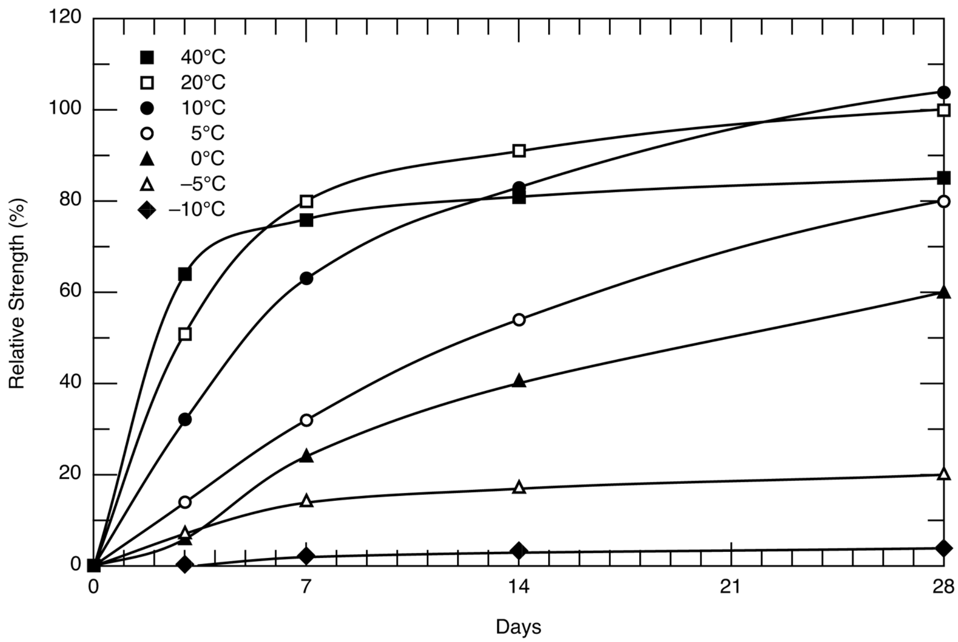

Figure 2. Effect of temperature on strength gain of concrete. All strengths are relative to the 28-day strength of concrete cured at 20°C (68°F). The strengths for 20, –5, and –10°C came from studies at CRREL, those for 40 and 5°C came from ACI (1988), and that for 0°C came from Timms and Withey (1934).

However, if concrete freezes after it has set up but before it has attained much strength, it can lose half its potential strength. Figure 2 shows that the three-day strength of –5°C concrete is less than 15 percent that of reference concrete cured at 20°C and that no strength developed at –10°C. If frozen at an early age, concrete never regains full strength. This is the situation to avoid. To avoid problems, the American Concrete Institute (ACI) (1988) instructs that concrete must be cured at 5°C or above. Not because hydration ceases below this temperature, but because it occurs so slowly. For example, concrete cured at 0°C is not harmed by the cold but it would take well in excess of 28 days to attain full strength (Fig. 2). Therefore, anything that can be done to help concrete gain strength faster during cold weather can reduce the duration of cold-weather protection, speed up production and, in effect, reduce the in-place cost of concrete.

Before discussing how current practice accommodates cold weather, it is important to note that it is becoming more common in general construction to use blended cements, such as fly ash, blast furnace slag, silica fume, and Type I/II cement. The driving force is a need to provide economy through improved

workability, reduced water/cement ratio, and reduced porosity of the concrete, in addition to providing cheaper cements applicable to a wider temperature range. Other reasons for using blended cements are to reduce adverse chemical reactions with aggregate and to increase concrete durability. Besides providing these benefits, blended cements often reduce heat of hydration, which can be advantageous for massive structures and for hot-weather construction to reduce temperature rise, but it is a serious drawback for cold-weather concreting when it comes to early-age strength gain. A recent study documented how Type F fly ash can slow down the development of strength in concrete. As can be seen in Figure 3, the curve for strength gained by concrete made with fly ash and cured at 20°C looks much like the curve for concrete made with Type I cement cured at 10°C in Figure 2. Essentially, the fly ash made the concrete behave as if it had been cured in cool weather. The other cements affect concrete similarly. Because blended cements can delay early-age strength development in concrete, they are not recommended for winter concreting unless heated enclosures are available.

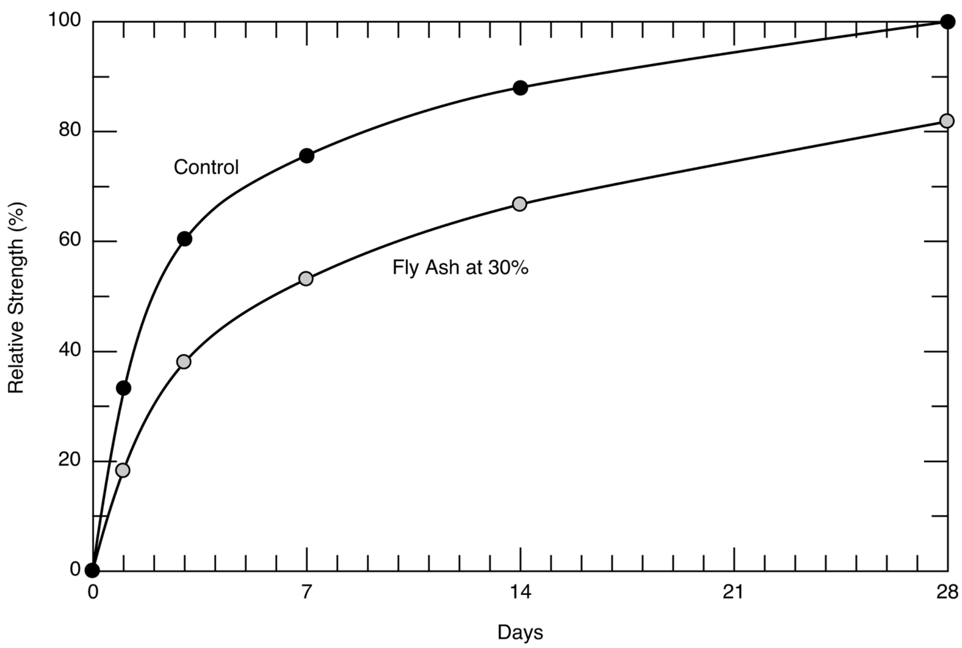

Figure 3. Using Type F fly ash as a cement replacement is not recommended for winter concreting if early strength gain is needed. (After Malhotra 1987.)

Cold-weather practice accommodations

Cold weather places serious constraints on today's concreting operations. As temperatures drop, concrete sets more slowly, takes longer to finish, and gains strength less rapidly. If temperatures dip too low, the risk is that the mix water will freeze, leading to irreparable damage. According to ACI (1988), concrete that is mixed and maintained above 5°C will allow construction to stay on schedule and freezing will not be a problem. Below that temperature, special precautions are necessary.

ACI provides several options to consider when the weather turns cold. The most common option is to use an ordinary summer mix, heat it so that it is delivered to the construction site warm, thaw the substrate upon which it is to be placed, and protect the concrete with insulation or heated enclosures while it cures. If it is necessary to accelerate early-age strength gain, ACI suggests adding extra cement to the mix or replacing the normal cement with high-early strength cement. Alternately, using a set-accelerating admixture, the equivalent of 2 percent calcium chloride ($CaCl_2 \cdot 2H_2O$) by cement weight, provides roughly the same benefit as does using more cement or high-early strength cement. Each option, as seen earlier, essentially compensates for a 10°C drop in temperature. Exercising any of these options is dictated by the benefits and by the costs involved. Sometimes, doing nothing and waiting for the return of warm weather is the best option.

If work cannot be halted, current cold-weather protection methods protect fresh concrete from freezing and ensure adequate strength gain to continue construction. This protection, however, can be expensive. Depending on the level of cold-weather protection employed, the surcharge—comparable to ready-mix concrete placed in the summer, which requires no protection—can range from a few percent, for heated mixing water, to over 100 percent, for heated enclosures (Fig. 4). Thus, cold weather can more than double the cost of concrete. Estimates place the annual cost of winter protection in the U.S. concrete construction industry at $800,000,000 (*Civil Engineering* 1991). An admixture that would lessen this expense would be an economic benefit.

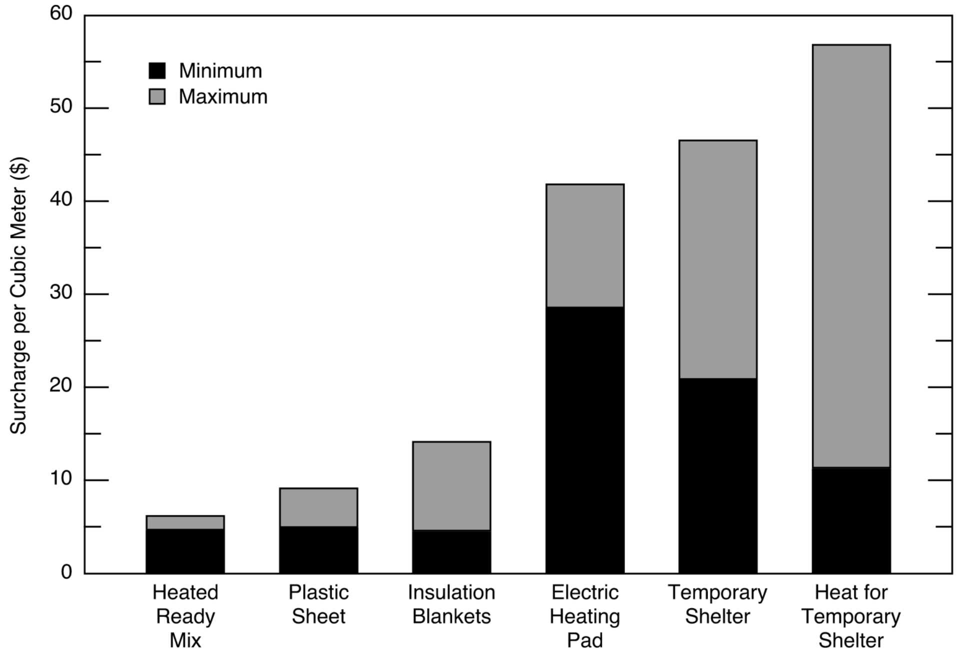

Figure 4. The surcharge to each cubic meter of concrete depends on the level of protection used. For comparison, a cubic meter of 34.5 MPa ready-mix concrete cost $78.29 in 1995 (Means 1993).

3 TEST PROGRAM

The objective of this study was to continue the investigation of commercial admixtures that was initiated in 1997 and that was further amplified in a report published in 2001 (Korhonen and Orchino 2001). The 2001 study advanced the state of the art by exploring a specific suite of admixtures aimed at allowing concrete to gain acceptable strength down to –10°C. The 2001 study nearly met that goal by lacing concrete with high dosages of set accelerators combined with water reducers. However, the resulting concrete, though it achieved freezing points down to –9.2°C, set up so rapidly, because of the high dosage of accelerators, that it would have had to be placed within minutes after the admixtures were added into the concrete, making it impractical for general field application. Nevertheless, the 2001 study established that off-the-shelf admixtures could protect concrete from freezing while promoting strength gain, all the while staying within approved dosage limits. More work was needed to solve the problem of early setting. Therefore, this study was designed to be less dependent on accelerating admixtures, and, as a consequence, the admixtures list was expanded to include corrosion inhibitors and shrinkage reducers.

As with the 2001 investigation, the benchmark of success for the admixtures in this study was the performance of reference concrete cured at 5°C. That is, any combination of admixtures, within approved dosages, that allowed concrete cured at subfreezing temperatures to gain strength as rapidly as reference (admixture-free) concrete cured at 5°C would be judged a success, provided the admixtures did not cause the concrete to set too rapidly or, equally important, not too slowly. It was arbitrarily established at the start of this study that the initial setting time of concrete made with admixtures and cured at low temperature would not be more than twice that of control concrete cured at room temperature.

Admixtures

Like the 2001 investigation, neither the manufacturer nor its product names are disclosed in this report. Because it was established in 2001 that commercial admixtures, when used alone within recommended dosages, provide little freeze protection on their own, individual admixtures were not evaluated for their effect on freezing point depression this time. Rather, a suite of six admixtures was selected: some for their ability to reduce the amount of mixing water, others to provide a modest increase in the rate at which cement hydrates, and in total to depress the freezing point of the concrete. By cutting back on the dosage of accelerator, compared to what was used in 2001, and by adding other non-accelerating admixtures, it was hoped that the combination of admixtures

selected for this study would allow the mix to remain workable for a reasonable period and be resistant to freezing without causing other problems.

The admixtures chosen for this study and shown in Table 2 either met the requirements of ASTM C 494 or were commercial products otherwise accepted by industry practice. As mentioned above, each admixture served a specific role. The Type A water-reducing admixtures were used because they can reduce the quantity of mixing water without reducing the workability of the concrete. They do this by breaking up the cement clusters that normally form in fresh concrete, with the result that the mixture retains its mobility, even at low w/c ratios. The benefit to using a water reducer is that less mixing water needs to be protected from freezing. Type A water reducers typically reduce water demand by at least 5% while Type F high-range water reducers reduce water demand by more than 12%—sometimes more than 25%. Mid-range water reducers, for which there is not an ASTM category, reduce water demand between these two limits. The downside of water-reducers is that they delay both initial setting times and early-age strengths.

Table 2. Suite of admixtures used in all but the control mortar.

Type	Dosage*
A	50
A & F	40
C	100†
E	60
Corrosion inhibitor	100
Shrinkage reducer	75

* Percent of maximum dosage printed in manufacturer's product literature.
† Equivalent to 2% $CaCl_2$ by weight of cement.

Type C accelerating admixtures are chemicals that are added into concrete during mixing to reduce the time of setting and to speed up strength development. Many chemicals are known to perform as accelerators, calcium chloride being the most popular. However, though calcium chloride is the point of comparison for all other accelerators, it causes embedded steel to corrode and, thus, it is being used less frequently in today's construction. Nonchloride accelerators are available, but their weakness is that they require higher dosages to match calcium chloride in effectiveness and, consequently, are more expensive. Accelerating admixtures are used in cold-weather concreting operations to reduce the time that concrete needs to be kept warm.

Type E water reducing and accelerating admixtures, as the name implies, provide both water reducing and accelerating cure properties to the concrete. Typically, manufacturers promote this category of admixtures for low-temperature concreting.

The final two admixtures used in this study, a corrosion inhibitor and a shrinkage reducer, were included not for their implied properties, but for the extra freezing point depression that they impart to the concrete. Their other attractive feature is that they are supposed to be set-neutral, neither causing cement hydration to speed up or slow down.

Mortar

To be consistent with the 2001 study, non-air-entrained* mortar, instead of concrete, was used to evaluate the performance of the admixtures selected for this study. Mortar is similar to concrete in that it contains cement, aggregate, and water, but it differs from concrete in that it contains only fine aggregate. (It was learned subsequent to this study that water reducers do not affect the workability of mortar the same way that they affect the workability of concrete. Thus, the flow measurements, used as the indicator of workability of mortar, presented in this report may not represent likely outcomes for concrete.)

Unlike 2001, in which a single mortar consisting of one part Type I portland cement to 2.8 parts ASTM C 33 (ASTM 1997b) sand was used, this study used four mortars. The idea was to illustrate the effect of cement content on time of setting and freezing point depression. Each mortar was designed to simulate the mortar fraction of one of four concrete mixtures, each containing a different cement factor. To simulate the mortar fraction of concrete, it had to be acknowledged that, in concrete, cement paste coats both coarse and fine aggregate and fills interstitial voids whereas, in mortar, it coats only fine aggregate in addition to filling voids. Thus, the sand/cement ratios could not be identical for both types of mixtures. Table 3 illustrates the resulting differences and shows the mix proportions used for each mortar used in this study. (In comparison, Tank and Carino [1991] used the coarse aggregate to cement ratio as the ratio for sand to cement when designing mortar from companion concrete. This ratio is included in Table 3 for reader interest.)

* Non-air-entrained mortar was used intentionally to determine the effect additives have on strength development without having to contend with variability of entrained air. Entrained air is recommended for field applications.

Table 3. Comparison of mortars developed from each of the concrete mixtures shown below. Each concrete mixture had a slump of approximately 80 mm and each mortar attained a flow of 120.

Mixture Type	Cement (K/m^3g)	W/C Ratio	Sand/Cement Ratio	Coarse Aggregate/ Cement Ratio
Concrete		0.52	3.09	
Mortar	308	0.46	3.16	3.5
Concrete		0.48	2.38	
Mortar	364	0.43	2.45	2.97
Concrete		0.44	1.88	
Mortar	420	0.36	1.95	2.57
Concrete		0.40	1.52	
Mortar	476	0.34	1.59	2.27

All mortars were mixed in a Hobart mixer according to ASTM C 305 (1987) at room temperature. All but the high-range water reducers were added into the mixing water, which was the first item placed into the mixing bowl. Once the water and admixtures were placed into the bowl, the mixer was run at low speed for 30 seconds while the cement was added to the water. Mixing was stopped, the sides of the bowl were scraped down, and the mixer was run for another 45 seconds while sand was added. The mixer was stopped for a minute and a half before the high-range water reducer was added with the mixer running at medium speed for the final minute. The water reducer was added during the first 15 seconds of the final mixing period. (Several trials were conducted to determine the optimum time to add the water reducer to the mix. Neither adding the water reducer immediately with the total mixing water nor delaying addition with one-third of the mixing water after the initial 30-second mixing period was as effective at increasing the mobility of the mix as was adding the admixture once all the aggregate, cement, and total water were partially mixed.)

Sample preparation and curing

Once mixing was completed, the mortar was cast into 50.8- × 101.6-mm plastic cylindrical molds, vibrated on a table to ensure consolidation, capped with plastic lids, and stored in rooms maintained at 20, –5, –10, and –20°C. All samples were placed into the temperature-controlled rooms within 30 minutes after water first contacted cement. Figure 5 shows how quickly the cylinders cooled in the various rooms. In this particular test, the center temperature of the cylinders reached 0°C within 7 to 55 minutes, depending on the temperature of the coldroom. It can be safely assumed that none of the mortars reached initial

set, let alone final set, before freezing occurred. They remained sealed until being tested for strength.

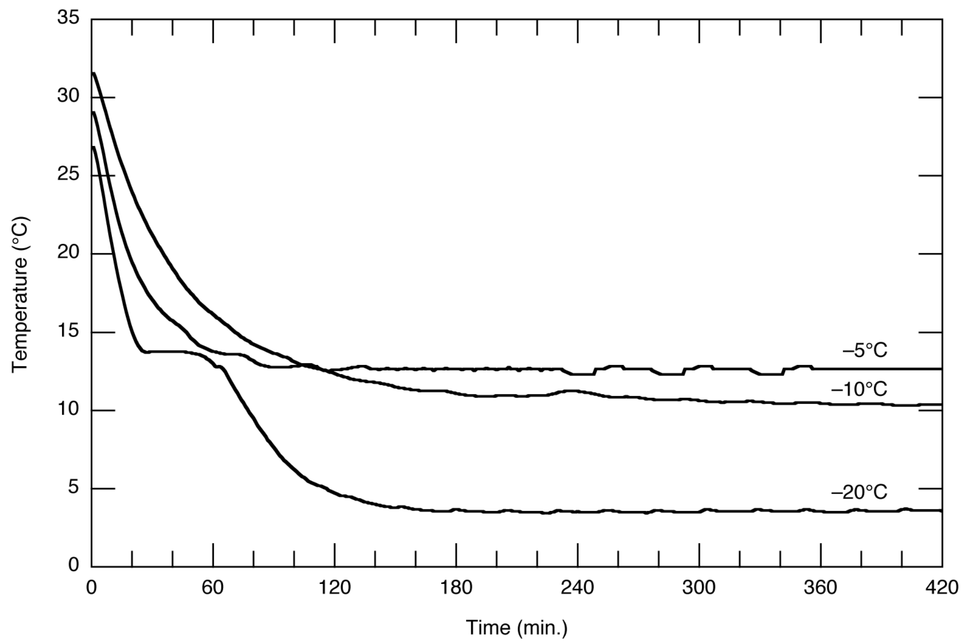

Figure 5. Typical cooling curves for 50.8- by 101.6-mm cylinders of fresh mortar. In this case, the rooms were maintained at approximately –3, –7 and –18°C.

Test procedure

The six commercial admixtures shown in Table 2 were tested for their collective effect on workability (flow), time of setting, freezing point depression, and strength gain of the mortars shown in Table 3. Flow tests were conducted according to ASTM C 109 (1989b), setting times according to ASTM C 403 (1981), freezing points from temperatures recorded from a thermocouple embedded into cylinders of fresh mortar placed into a coldroom, and compressive strengths were obtained according to ASTM C 39 (1997c). Prior to testing the cylinders for compressive strength, they were removed from their curing rooms, stripped from their molds, allowed to warm to 5°C (if necessary), and then capped with unbonded neoprene held within steel-retaining cups conforming to ASTM C 1231 (1997d).

4 RESULTS AND DISCUSSION

Standard practice places no limit on the number of admixtures that should be used in concrete, just on individual amounts—and then, these amounts can be exceeded case by case. Therefore, the approach was to use as many admixtures as needed to develop a mortar resistant to freezing down to –10°C, that would retain its workability for a reasonable period, and that would cure while at that temperature. The individual admixtures used in this study were limited to the maximum dosage recommended by the manufacturer. Since the initial laboratory investigation demonstrated that set accelerators combined with water reducers performed well, except for workability problems, this study used both types of admixtures, along with the addition of corrosion-inhibiting and shrinkage-reducing admixtures.

Freezing point

Freezing points were determined by embedding thermocouples into 50.8- by 101.6-mm cylinders of fresh mortar placed into a –20ºC room. Figure 6 shows a typical cooling curve for an antifreeze mortar compared to that of tap water. The freezing point on each curve is identified as the approximate location where the slope of the cooling curves changed. Note that at that location, both cylinders slightly undercooled before suddenly warming up a bit. The lowest undercooling represents the temperature at which ice spontaneously nucleates, releasing the latent heat that warms the system, while the highest rebound temperature defines where ice grows (i.e., the freezing point). The constancy of temperature for the tap water suggests that tap water has only one freezing temperature while the mortar, a solution of alkalis and other chemical additives, does not freeze at one temperature. The shape of the curve that follows the slope-change illustrates this where the tap water continues to freeze at –0.1°C while the mortar needs progressively lower temperatures to promote additional freezing below –7.1°C. In this study, –7.1°C would be considered the freezing point of the mortar.

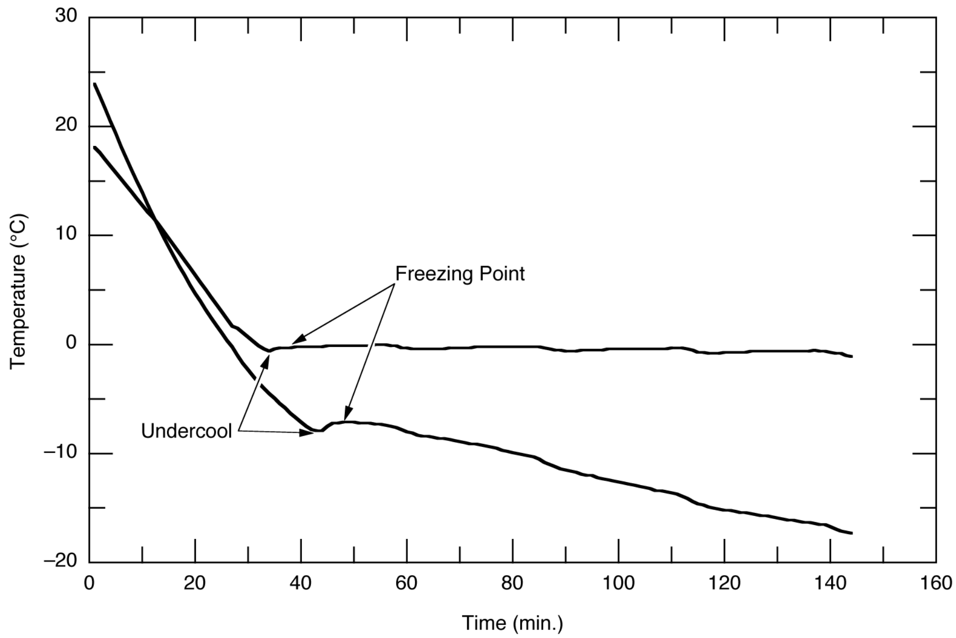

Figure 6. Cooling curves for tap water (top line) and an antifreeze mortar.

Table 4. Freezing point measurements.

Control Mortar	°C	Antifreeze Mortar	°C
517C	–1.5	517	–6.4
611C	–1.4	611	–7.6
705C	–1.7	705	–9.2
799C	–1.8	799	–10.1

Results for the four mortars containing the admixtures in Table 2 along with those of their companion control mortars are presented in Table 4. Before we consider the antifreeze mortars, it is interesting to note that the four control mortars all froze at essentially the same temperature. This outcome is not immediately intuitive as one might expect higher alkali concentrations in lower w/c ratio mixtures and, consequently, lower freezing points. However, it did not seem to matter that although the w/c ratio ranged from 0.52 to 0.40, all mortars acted alike. What is more, other studies have pointed out, similarly, that mortars (concrete) made without chemical additives typically have one temperature at which nearly all of the mixing water freezes (Mironov 1997). Corroborating this are cooling curves, obtained elsewhere, that show that once freezing occurs in

control mortar the temperature of the mortar remains constant for an extended period (Korhonen 1999); it does not gradually fall off as shown for the antifreeze mortar in Figure 6. Thus, changing the w/c ratio will not provide increased freeze protection and, once freezing initiates in admixture-free concrete, a high percentage of the mixing water will immediately turn into ice and structural damage is likely.

The four antifreeze admixtures froze at temperatures that were significantly lower than those of the control mortars, but not all froze at the same temperature, even though the admixtures were dosed at the same rate in each of the four mortars. The reason that the freezing points varied from mix to mix was that the admixtures were dosed based on the amount of cement and not the amount of water. This meant that the mortar with the highest cement factor coupled with the lowest w/c ratio would have the highest concentration of admixture in its mixing water and, thus, the lowest freezing point. It did. Figure 7 illustrates how the freezing point varied with the concentration of admixture found in the mixing water of each of the four mortars. As can be seen, the freezing point is linearly proportional to the amount of admixture dissolved in the mixing water, and going to higher cement factors yields lower freezing points because less water is needed to maintain workability. The freezing point can be raised or lowered from what is shown in Figure 7 by either increasing or decreasing the number of admixtures or, alternately, by increasing or decreasing the dosage of individual admixtures. What is apparent from this information is that increasing the cement content, in effect decreasing the w/c ratio, allows the concrete to resist lower and lower temperatures without changing the admixture dosage rate.

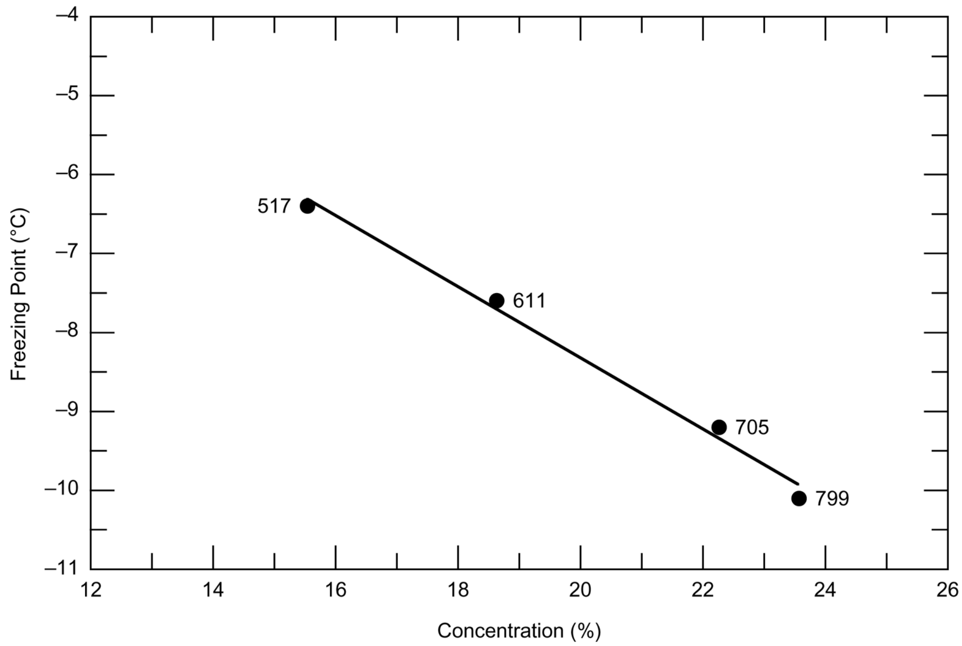

Figure 7. Freezing point versus concentration of admixture (total solids) by weight of water for each of the four mortars shown in Table 3.

Table 5. Initial and final times of setting.

Mortar	Initial Set (Hr)	Final Set (Hr)
517C*	3.8	5.1
517†	5.0	6.2
611C	3.6	4.9
611	4.25	5.2
705C	3.45	4.5
705	3.25	4.5
799C	3.2	4.2
799	3.4	4.1

* Control mortar
† Antifreeze mortar

Time of setting

The initial and final times of setting for both the control and the antifreeze mortars are given in Table 5. The goal was to create a mixture that would not set too quickly. The decision was to test all admixtures at room temperature to

represent the most severe condition. Anything cooler would slow down the setting time. Tests were not done at the low temperatures the mortars were designed for, but this should be done in future testing to determine that the mortars do not set too slowly. As the data in Table 5 suggest, none of the anti-freeze mortars appeared to set too quickly. If anything, they were a bit slow. Therefore, it seems reasonable to assume that quick setting should not be a problem when the mortar temperature is as high as 20°C. However, the effect of having large quantities of concrete in a mixing truck where heat rise may be even more severe than it was with the smaller laboratory batches needs to be investigated.

Compressive strength

Three samples of each mortar from each of the four curing rooms were tested in uniaxial compression according to ASTM C 39 at 3, 7, 14, 28, and 56 days. Each sample was capped with unbonded neoprene held within a steel-retaining cup according to ASTM C 1231. The samples from the coldrooms were allowed to thaw to 5°C before being tested. The results are presented in Figure 8 and in Table 6.

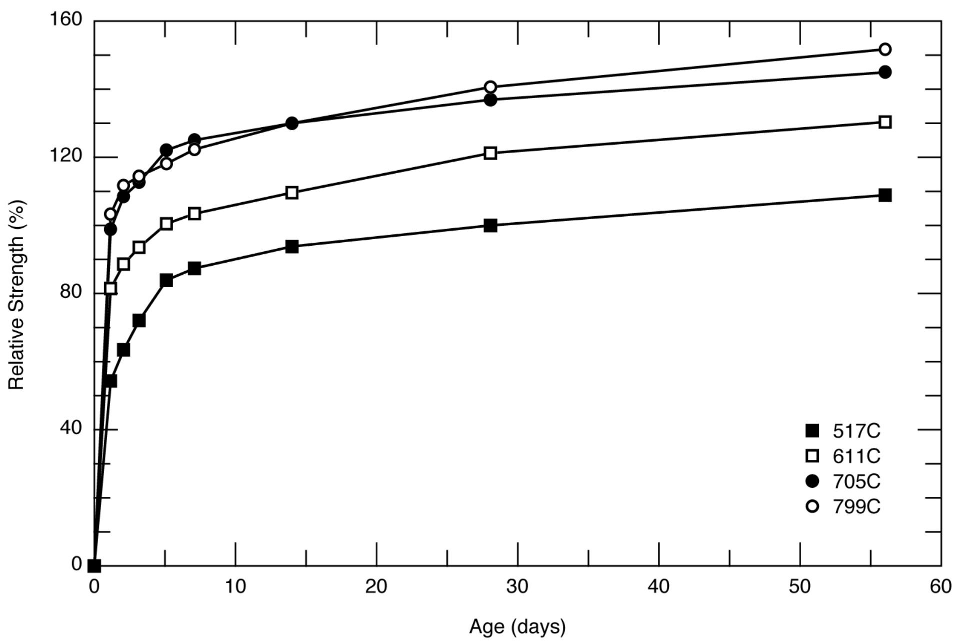

Figure 8. Compressive strength relative to the 28-day strength of mortar 517C. All samples cured at 20°C.

Table 6. Relative strengths of mortars cured at various temperatures for 14 days, then at 20°C for 42 days. The strengths of a given mortar are based on the 28-day strength of that mortar cured for 28 days at 20°C. Typically, control concrete cured at 5°C gains 30, 50, 80, and 100 percent of its 28-day room-temperature strength in 7, 14, 28, and 56 days, respectively. Each relative strength represents the average of three tests.

20°C						–5°C					
	Days						Days				
Mortar	**3**	**7**	**14**	**28**	**56**	**Mortar**	**3**	**7**	**14**	**28**	**56**
517C*	72.0	93.0	93.7	100.0	108.7						
517†	62.3	81.2	92.0	105.9	122.2	517	6.7	36.9	61.9	117.0	124.9
611C	77.0	84.9	90.1	100.0	107.0						
611	79.1	102.1	110.9	121.0	129.9	611	3.8	44.7	79.3	131.6	155.2
705C	82.7	91.5	95.0	100.0	106.2						
705	94.5	105.4	114.5	124.9	137.0	705	na	na	na	124.8	134.7
799C	81.4	87.1	92.4	100.0	108.2						
799	94.8	108.5	118.9	130.3	144.1	799	na	na	na	124.1	131.0
–10°C						–20°C					
	Days						Days				
Mortar	**3**	**7**	**14**	**28**	**56**	**Mortar**	**3**	**7**	**14**	**28**	**56**
517	0.5	5.8	19.9	82.4	88.0	517	0.3	0.7	0.9	59.4	71.6
611	1.2	12.1	30.4	110.6	130.1	611	0.5	0.9	0.9	63.6	76.7
705	na	na	na	123.7	134.4	705	0.6	1.0	1.4	80.2	92.8
799	na	na	na	120.6	141.4	799	0.9	1.0	1.4	77.1	90.4

* Control mortar

† Antifreeze mortar

Na: Data not available; malfunction in data acquisition system.

Before discussing the low-temperature performance of each admixture, it is perhaps interesting to note the performance of the control and antifreeze mortars at room temperature. For the control mortars, Figure 8 indicates that the ultimate strength attained by each mortar increased with cement content. Specifically, 611C, 705C, and 799C attained 21, 37, and 41 percent, respectively, more strength than did 517C after 28 days of curing. This trend continued through 56 days. This is no surprise nor is it surprising to see that Table 6 shows that each control mortar had roughly the same percentage increase in strength at each age. That is, each control mortar gained approximately 75, 85, 92, 100, and 108 percent of its 28-day strength in 3, 7, 14, 28, and 56 days. Therefore, though the amount of cement used in a mix affected absolute strength, it did little to change the way strength was developed. (For comparison, concrete has been known to gain about 75 percent of its 28-day strength in seven days when cured at room

temperature [Korhonen 1999].) Interestingly, the admixtures, which contain accelerators, did not cause the 517 mortar cured at room temperature to gain strength more rapidly than its control did over the first 14 days, as they did for the other three mortars. It is not clear why this happened. However, it is clear that the admixtures do not interfere with the development of long-term strength, as each mortar ultimately became stronger than did the control. This is an important finding.

At the three low temperatures, the admixtures forced the mortars to develop more strength in a shorter period than they otherwise would. Typically, mortar made without admixtures gains less than 15 percent of its potential strength in seven days when cured at –5°C, less than 2 percent when cured at –10°C, and essentially no strength when cured at –20°C (Korhonen 1999). Clearly, the four mortars made with the admixtures outperformed these measures at all three low temperatures. The benchmark of success, set at the start of this study, was to gain strength at least as rapidly as control mortar cured at 5°C. This means that the antifreeze mortars, when cured at low temperature, should gain at least 30 and 50 percent potential strength at seven and 14 days, respectively (Korhonen and Orchino 2001). All mortars easily exceeded those values at –5°C. In fact, they gained strength as if their internal temperatures were somewhere between 10 and 20°C.

Though no data are shown for the 705 and 799 mortars for 3, 7, and 14 days, the fact that they had lower freezing points than 517 and 611 and that they outgained their control mortars at 28 and 56 days strongly suggests that they performed equally as well as the 517 and 611 mortars at seven and 14 days. At –10°C, the 517 mortar failed to meet the benchmark by a wide margin while the 611 mortar came close to being successful. For reasoning similar to that given for the –5°C data, it is strongly suspected that the 705 and 799 mortars succeeded at –10°C. The mortars were not expected to succeed at –20°C because their freezing points did not go that low. Therefore, based on the original definition of success, the "freezing protection temperature"—the lowest temperature at which mortar develops strength at an appreciable rate—is –10°C for 705 and 799. For 517 and 611, the freezing protection temperature is somewhere between –5 and –10°C.

If early age strength is not critical, another way to judge success is whether the mortar, cured at low temperature, will eventually attain full strength when thawed. The practicality of this is that a concrete structure could be cast and left alone during severely cold weather, and, when the weather turns warmer, the structure would begin to develop strength. Using this yardstick means that all but the 517 mortar succeed at –10°C.

Another possible measure of success is to consider that hot summertime conditions can render concrete up to 25 percent weaker at the 56-day mark compared to concrete cured at cooler room temperature conditions (Korhonen and Orchino 1999). Holding concrete cured at subfreezing temperatures to these expectations qualifies all but the 517 mortar down to –20°C. The 56-day compressive strength ranged from 76.7 to 92.8 percent potential strength. Thus, the "damage protection temperature"—the lowest temperature at which strength gain may not be significant but from which the concrete can adequately recover—is –20°C for 611, 705, and 799, and is somewhere between –10 and –20°C for 517.

5 CONCLUSIONS

None of today's admixtures satisfy the minimum strength requirements established at the beginning of this report for concrete cured at subfreezing temperatures. Part of the problem is that individual admixtures are not dosed into concrete at high enough concentrations to depress the freezing point of fresh concrete by more than a degree or two. This is not enough protection to preclude the need for thermal protection during cold weather. Simply increasing individual admixture dosage rates is not acceptable if current limits are to be honored. Thus, current practice limits cold-weather protection to insulation, supplemental heating, and tenting. Though existing data show that using high doses of certain combinations of chemicals will allow fresh concrete to gain strength at below-freezing temperatures without causing detrimental effects to the final product, the other part of the problem is that standards do not exist to support the development of new low-temperature admixtures. Unless something is done to raise the awareness to this new approach to winter concreting, the state of practice will remain pretty much as it has been since the 1930s.

This study evaluated the efficacy of using existing admixtures in combination as a way to increase total admixture dosage without violating individual dosage limits. The idea was that, alone, no admixture could sufficiently depress the freezing point of the mixing water and accelerate cement hydration, but in combination they might form an admixture that could satisfy both functions. And, if used within current dosage limitations for individual admixtures, the resulting blend of admixtures should be acceptable without the need for new standards because there are no restrictions on the number of admixtures that can be used in concrete. Thus, an admixture could be developed that satisfied current standards and that would allow the cold-weather concreting envelope to legitimately extend into the subfreezing for the first time.

This study showed that admixtures used in today's concrete could be combined to allow fresh mortar cured below 0°C to gain strength as rapidly as control mortar cured at 5°C and above. Of equal importance was the finding that the admixtures chosen for this study did not interfere with the development of strength when the mortar was warm. Using six commercial off-the-shelf admixtures produced mortars with freezing points ranging from –6.4°C down to –10.1°C. All mortars nearly matched the strengths expected from control mortar cured at 10°C. For those mortars with freezing points below –9°C and cured at –10°C, their strengths exceeded that attained by control mortar cured at 5°C. By allowing the concrete to lose some strength, comparable to what happens now in summertime conditions, the mortars with freezing points at or below –7.6°C were

able to nearly fully recover strength when cured at temperatures as low as –20°C for up to 14 days. Unlike the results of the 2001 study, where the mortar set too rapidly to be of practical value, this time the mortar behaved much like control mortar in respect to time of setting. The admixtures performed well in mortar under controlled laboratory conditions. They should now be tested in concrete under field conditions.

6 NEEDED RESEARCH

Commercial off-the-shelf admixtures have limitations. For low-temperature applications, these limitations center on the water that is contained in the admixtures. If all ingredients for a concrete mix were to be batched at a single point, it would be an easy matter to adjust the amount of mixing water to account for the water contained in the admixtures and all would be well. My experience, however, has been that it may not always be possible to incorporate all of the admixtures into the concrete at the start of mixing. Sometimes it is necessary to delay one or more of the admixtures, particularly the accelerators, until later so that the concrete does not stiffen before it can be placed. However, delaying the addition of one or more of the admixtures creates a potential problem in that the initial concrete mix is now drier. It seems that when the w/c ratio is lowered below 0.42, the concrete has no slump and little entrained air. Fortunately, using plasticizers can alleviate most of these problems. Although added to increase fluidity, plasticizers also increase the rate of slump loss that occurs in fresh concrete. In itself, slump loss of concrete in transit to a job site may not be a problem, provided that the mix can be returned to being workable in time for placement. If the decrease in workability during transit is not caused by the partial hydration of cement, the concrete should be able to be brought back to being workable once the last admixture is added. (The ideal situation would be to use a dry accelerator. This would allow all of the mixing water to be added into the mix at the ready-mix plant and the dry ingredient could be added at the job site just prior to placement.)

Although the results from the testing done on small quantities of mortar suggest that rapid setting and slump may not be a problem from concrete made with the admixtures used in this study, the effect of having large quantities of concrete in a mixing truck where heat rise may become an issue needs to be investigated. It is recommended that moderately sized batches (about 3 m^3) be tested for changes in slump, air content, mix temperature, and unit weight as a function of time, starting as soon as the concrete is mixed. Primarily, work needs to be done to determine how to administer the admixtures into concrete. Should they all be added at the batch plant or should part of them be delayed until the ready-mix truck reaches the construction site? As alluded to above, the answer to this depends on how well the concrete mixture retains its workability at any stage of the process. Until this is done, it is not recommended that the results of this study be applied directly to field applications.

Because slump loss does not necessarily indicate anything about cement hydration, especially when plasticizers are used, it is recommended that the time of

setting be measured for each mortar cured at its lowest working temperature. This would determine whether the construction schedule would be unduly long in cold weather.

Finally, although all admixtures have individually passed a battery of tests to prove that they cause no harm to the concrete, little is known on how they affect the freeze–thaw durability of concrete when used in combination. Thus, the admixture combination used in this study should also be evaluated for its affect on the freeze–thaw durability of concrete.

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14. ABSTRACT

Cold weather places serious constraints on today's concreting operations. As temperatures drop, concrete sets more slowly, takes longer to finish, and gains strength less rapidly. If temperatures dip too low, the risk is that the mixing water will freeze, leading to irreparable damage. Current guidance limits cold-weather protection of fresh concrete to insulation, supplemental heating, and temporary shelters to keep the concrete at or above 5°C throughout the curing process. This paper studies the use of commercial admixtures in combination with one another to depress the freezing point of the mixing water and to allow the concrete to gain strength at below-freezing temperatures without thermal protection.

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COLD REGIONS RESEARCH AND ENGINEERING LABORATORY, 72 LYME ROAD
HANOVER, NEW HAMPSHIRE 03755-1290

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